MDA From Helional: A Two Part Synthesis

Preface to Part 1

Part 1 is the "one pot" aldehyde (helional) to amide (helionalamide or MMDPPA). A few reactions occur in a few steps, but just keep in mind that from the aldehyde you first form a nitrile intermediate (by way of an aldoxamine), and then the nitrile is selectively hydrolyzed to the amide.

First hydroxylamine reacts with helional and forms an aldoxamine which is dehydrated to a nitrile with heating, then hydrogen peroxide reacts to form the amide. Let cool to precipitate out the amide, wash with water, and finally recrystallize in IPA for the final amide product, which will be made into MDA in the second part.

There is no need to isolate the aldoxime and then preform a Beckmann rearrangement with nickel acetate as a catalyst ("twodogs method"). All of the reactions occur without a need to isolate any of the intermediate compounds.

Chem Info

Helional : 3-(1,3-Benzodioxol-5-yl)-2-methylpropanal or "**The Aldehyde**", $C_{11}H_{12}NO_3$
Mol. weight: **192.214 g/mol** (freebase), Density: **1.162 g/cm³**, boiling point: 282 °C
Colorless liquid with a pleasant, faint smell of flowers. Used in perfume. Have a waft of it!
(CAS Nr.: 1205-17-0) Readily soluble in DMSO, but not water. Might have a yellow tinge.

Hydroxylamine: (hydroxyammonia), NH_2OH
Use the stable HCl salt form (Hydroxylammonium chloride), chemical formula $[NH_3OH]Cl$, with a
Mol. weight of **69.49 g/mol**
Powder, fine crystals, that dissolve well in water or DMSO.

Hydrogen Peroxide: H_2O_2
Mol. weight of 34.014 g/mol, but use about a **30% solution** in water, with a molarity of 9.79M, or **9.79 mol/ Litre.** Calculate the molarity exactly depending on the concentration of your peroxide.
Liquid, miscible with water, DMSO.

DMSO: dimethyl sulfoxide, C_2H_6OS
Use as the **solvent** for the reaction, harmless, but the smell of garlic gets strong and annoying when heated.
Melting point of 19 °C (so likely you will find crystals in a bottle, melt before reacting) Boiling point: 189 °C

Sodium Hydroxide or NaOH
Mol. weight of ~**40 g/ mol**, will need lots in both parts of the synthesis to MDA.

Product is:
MMDPPA or helionalamide or "**The Amide**" (α-methyl-1,3-benzodioxole-5-propanamide, IUPAC: 3-(1,3-benzodioxol-5-yl)-2-methylpropanamide), $C_{11}H_{13}NO_3$
Mol. weight: **207.23 g/ mol**, (CAS Nr.: 858215-05-1), listed as soluble in DMSO 25 mg/ mL, but essentially insoluble in water, especially cold. Shiny crystals.

"Quick" outline:

- Mix 1:4 helional to DMSO in a beaker with ample room for foaming, with larger amounts think about how to mitigate the smell of DMSO, possibly use a three-neck flask or cover the beaker well.
- Add in about a 1.00 – 1.05 x molar amount of hydroxylamine HCl to helional, but do not add all at once, or only heat mildly, there is a temperature spike with a delay once the hydroxylamine starts to react. Make sure you weigh accurately, you want all of the hydroxylamine to react with none left that might potentially violently react with peroxide later on. (For **100 g helional, use ~36.5 – 36.9 g Hydroxylamine HCl**)
- Heat for 90 minutes at 90 °C, note colour change, after 90 minutes – 2 hours let solution cool to 70 °C, so when adding the first bit of H_2O_2, there is room for a temp. spike.
- Pre-basify the 30% or 35% H_2O_2 to a pH slightly above 7. Use somewhere 4 – 5x volume amount of H_2O_2 to helional in grams . But a bit less should also work. (For **100 g helional use 400 – 450 mL 30% H_2O_2 or 350 mL of 35%**). This is 4 mol of H_2O_2 or 7.7x mol excess, however maybe only a 5x molar excess is needed? Most of the peroxide decomposes due to heating and does not take part in the reaction. Regarding the basicity: you want the peroxide to react at once when it hits the mixture but not too strongly. If you add acidic H_2O_2 it will not react, but once it becomes basic it could react all at once dangerously. As well avoid heating DMSO anywhere near it's boiling point, could be very dangerous in the presence of peroxide.
- Basify the **reaction mixture to pH 10.** The reaction does not run below pH 10, and is already too reactive above pH 10.3.
- Best to use 2 addition funnels, one for NaOH 15% solution, the other for H_2O_2, and a pH probe submerged the entire time in preparation for peroxide addition.
- Drip in H_2O_2 but the mixture will drop below pH 10, so drip in some NaOH solution to raise it. Find an optimal drip rate for both so that the **correct pH of 10 – 10.3** is maintained, and the correct **temperature of 85 – 90 °C** is held. High temperatures will hurt the yield, as will peroxide additions at too low of a temperature.
- Move your flask or beaker to cold water if you drip in too much H_2O_2 when the pH has been higher. This will cause strong foaming, and hissing along with a temperature spike. In this case a round bottom flask and a heating mantle is less appropriate as a wet flask can not be used on the mantle sleeve.
- Once done with peroxide addition let the reaction mixture cool to 50 °C, transfer to a pyrex plate (or larger beaker), dump in some cold water, or just let it cool slowly and transfer to a fridge.
- Suck dry at the pump in a Büchner funnel, and **wash with lots of cold water** to remove traces of MSM.
- Now there is an option of **recrystallizing in boiling water** and then recrystallizing in a mixture of mostly IPA and some water. Maybe you could just wash with water, then recrystallize in IPA as well.
- Suck dry again, and leave it out to dry fully, **yield should be ~70 – 80%** of the recrystallized product, just make sure that you do not use too much solvent for recrystallizations and do so carefully, as much product loss is possible in the final steps. Proper recrystallization in an Erlenmeyer flask.
- For the second step, the amide must be relatively pure. It should sparkle, and have very little visible yellow.

Procedure for a 100 g helional run:

1) Mix 87 mL (101 g) helional with 400 mL DMSO in a 2L beaker, Color dissolved is a light yellow. Calculate as **0.525 mol**. Turning on heating and magnetic stirring on a large hot plate. Might be able to get away with less DMSO, but a 4x grams of aldehyde to mL of DMSO is the rule of thumb.

2) Mix in 36.6 g Hydroxylamine HCl or **0.526 mol** (Use just a bit more than an equimolar amount to the amide). Weigh exactly, as any excess hydroxylamine will react strongly with the peroxide later on. So weighing now carefully might prevent a run-off reaction later on. The stuff is fine in salt form but it is dangerous in freebase form, NaOH will deprotonate it. But it seems to react fine in straight DMSO that has not been made basic, but with heating.

3) Exothermic with lots of vapour, **scent of garlic**. Colour darkens, darker yellow.

4) Mix up **21.5 g NaOH** in 140 mL dH_2O in a beaker and set aside. (**15% solution**)

5) More and more vapour, and smoke are released and the temp. rises to 110 °C when away from the beaker, after only a short period. Need to quickly transfer the beaker to cold water to cool. After addition of hydroxylamine watch the temperature. It will rise but only after a delay. So heat the beaker or flask only gently or add the hydroxylamine in portions.

6) Color starts to turn orange, let it spin for 2 hours once temperature is between **80 – 86 °C**. Turns to orange, then a murky red later on.

7) Pre-basify **450 mL of H_2O_2 35% solution**. This is about 5.23 mol, which is a large excess. Needed as most H_2O_2 is destroyed and does not react. Should be able to use a fair bit less. However the aqueous hydrogen peroxide helps keep the amide dissolved as well. Add NaOH carefully until you reach a pH of 7 – 7.5, measuring with a pH probe. Use about 0.5 - 1 g NaOH.

8) Once it has been spun for a few hours under heating, with a full darkening of colour, **basify your reaction mixture just above pH 10** with a portion of the 15% NaOH solution prepared beforehand. Adding the NaOH solution increases temp from 70 to 80 °C, and the reaction mixture changes quickly from a dark orange to a turbid brown- red, then clearer. The pH is 10.42 of the reaction mixture. This pH as well keeps the reaction from getting out of control.

9) Difficult to raise temp above 80 °C prior to the addition of H_2O_2. Set hot plate temp. all the way up to 154 °C. During the reaction with hydroxylamine, had set it at 130 °C to react at 85 °C.

10) The H_2O_2 solution is loaded in a **500 mL addition funnel** with a pressure equalizing side arm, but a regular addition funnel works as well. It is best to load the remainder of the 15% NaOH solution in a second addition funnel, and from a ring stand hang a pH probe.

11) When you are **dripping in the peroxide, the pH falls**, once it is too low, the reaction stops, so you must **keep adding NaOH**. The pH has to remain around 10 the entire time of peroxide addition, too high pH and it could react too quickly.

12) **pH decreases about 0.01/ second with 1 drop/ second of added peroxide** solution.

13) The reaction is very exothermic so little heating is needed once additions are begun above 80 °C. Keep a large bath with cold water if temperatures go above 90 °C. Easy to cool the beaker quickly this way.
Lots of foaming and fizzing, and other rude sounds especially when the pH is higher. Remember, the optimal range is between 10 and 10.3 pH. The optimal temperature is 85 – 90 °C. Find the optimal drip rate to keep steadily within this range.
Gamification, so Generation Z won't get bored cooking too quickly. It is fun guys.

14) Once done with peroxide addition, let the reaction die off as it cools to 50 °C or so, then you can dump in cold water, very soon the product should appear and fill the entire beaker or pyrex plate if you have transferred to one. Poured in about 300 mL water. Could have been too much, pour instead about 10% by volume of the reaction mixture.

15) Set in the fridge for a bit (overnight) and filter a bit in a Büchner funnel, **wash with fresh water** as well.

16) Use 800 mL of water to recrystallize the product, this is too much, heat in an Erlenmeyer flask adding boiling water, making sure it just dissolves just when the water is close to boiling. Let cool slowly as well. Above **80 °C the amide tends to dissolve** quicker.

17) Filter and dry somewhat again.

18) Do another re-x, but this time with 100 mL IPA + 400 mL dH_2O set to boiling, But this is too much water again, and the amide fully dissolves far below boiling (around 80 °C). Use a round bottom flask, and a heating mantle to heat up quicker compared to a hotplate and beaker. Next time I would use more IPA with less water, or all IPA for the second recrystallization.

19) Set in fridge, then freezer for a bit, filter, dry and weigh, end up with 96.992 g amide. However this is not the true yield. Turns out that the amide is slow to fully dry. The amount after leaving out for a few days in a glass dish with intermittent mixing is **77.9 g** of a sparkly amide with just a very light hint of yellow. Makes for **0.376 mol**, so about a **72% yield** from the starting helional.

Additional Info:
100 mL helional is 116.2 g, 100 g helional is 86.06 mL, 100 g is 0.52 mol
MSM(methylsulfonylmethane) is formed during the reaction from oxidation of DMSO, but that is easily washed away with water, its appearance is similar to helionalamide but most similar to shards of meth.

TLC monitoring of the reaction could be attempted with an **eluent of 1:4 EtOAc: Hexane**. However the DMSO would cause a huge streak and blob on the plate, so one would need to draw up an aliquot of the R.M. then dilute with a few drops of water and then add a few drops of EtOAc. Agitate to extract into the ethyl acetate, and then finally take an aliquot of that for TLC. Next time.

The reaction shouldn't give problems at a 300 g or 700 g scale, just need a larger beaker or flask and a longer addition time of peroxide…

This Part 1 is **Scarecrow's method**, originally posted in this thread:
hxxps://www.thevespiary.org/talk/index.php/topic,4280.msg40444.html#msg40444
The idea from this article:
hxxps://chemistry.mdma.ch/hiveboard/palladium/pdf/FACILE%20ONE-POT%20CONVERSION%20OF%20ALDEHYDES%20INTO%20AMIDES.pdf

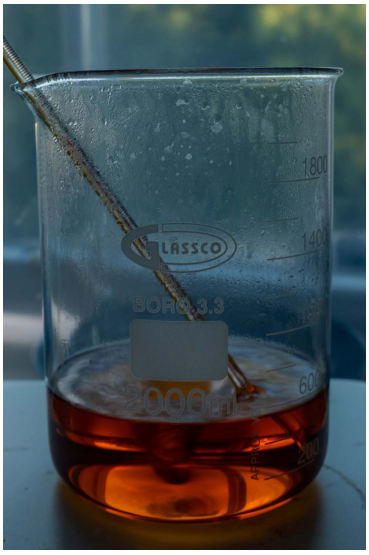
Finally, from the MMDPPA it is possible to make MDMA as well, see here:
hxxps://www.thevespiary.org/talk/index.php/topic,21767.msg54231648/topicseen.html#msg54231648

However, note that the procedure does not scale well. Best to make MDP2P from Helional and then proceed to MDMA if that what is you are after.
Like in this method:
hxxps://www.thevespiary.org/talk/index.php/topic,19177.0.html

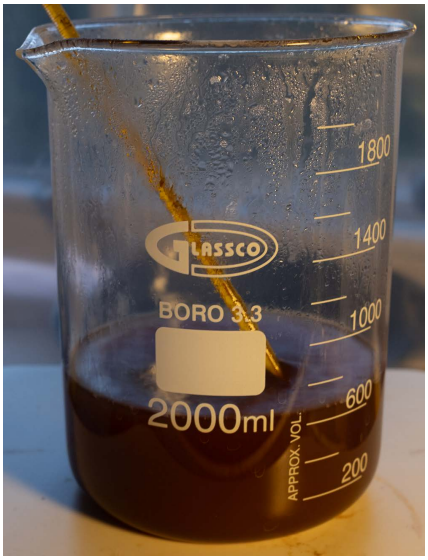
Just keep in mind that MDA is a superior compound.

Part 1 Photos:

Dissolve Helional in DMSO

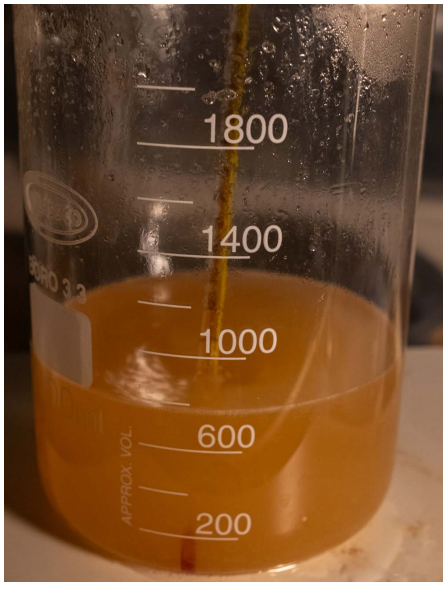


Hydroxylamine reacts with Helional and DMSO stinks



Reaction with hydroxylamine toward the end

Addition of hydrogen peroxide begins



H_2O_2 reaction, color change

End of H_2O_2 addition

Pour into a plate and pour cold water, product appears

Product is precipitated in the plate

Mostly white MMDPPA after recrystallizations

Preface to Part 2

Part 2 is a Hofmann rearrangement with NaDCC or Sodium dichloroisocyanurate as the halogen source. The unanimous verdict is that it works better than both TCCA (Trichloroisocyanuric acid) or Sodium Hypochlorite. Dissolves readily in water unlike TCCA and easy to handle with good yields.

As in Part 1, it is best to think of the reaction in two sections. Here a complex reaction takes places as well. First is chlorination with NaDCC in the cold to form an **N-chloroamide intermediate**. Then remove the formed cyanuric acid so it doesn't get in the way. For the second half, for the rearrangement itself, heat to form an **isocyanate intermediate** before forming a transient **carbamic acid** that decarboxylates into MDA freebase. There will be dark impurities, which can be dealt with in a number of ways (distillation or a few acid/ base extracts, cleaning with EtOAc and acetone), before we end with the final MDA HCl.

This can be a high-yielding reaction, and 80% is attainable, you just need to **let the reaction go slow,** and deal with it in two halves. It is not voodoo.

Chem Info

MMDPPA or helionalamide or "The Amide", written in all nooks and crannies of the internet that it must be of high purity for the reaction to go well, for a decent final yield.

NaDCC: Sodium dichloroisocyanurate $C_3Cl_2N_3NaO_3$
Will likely find it as the **dihydrate form** with a CAS Nr.: 51580-86-0 and a
Mol. weight of **255.98 g/mol** (dihydrate), Solubility in water is variously written as 22.7 g, 25 g, or **28 g/ 100 mL**. So thereabouts. It has two chlorine atoms, so we will use half the molar amount as The Amide. Sold as quick dissolving chlorine pool disinfectant under the name of "Di-chlor" in its pure form. TCCA being "Tri-chlor" but in chunks and sometimes with additives.

Product is:
MDA: 1-(2H-1,3-Benzodioxol-5-yl)propan-2-amine or **3,4-Methylenedioxyamphetamine** or "**The Amine**" $C_{10}H_{13}NO_2$, Melting point of the HCl salt is given variously from 180 – 181 to **185** to 188 °C. **pKA is 9.67**
Mol. weight: 179.22 g/mol (freebase), **215.68 g/mol** (HCl salt). The freebase is a colourless liquid at room temp, the HCl salt forms rocky dense crystals out of evaporated water or alcohols.

Miscellaneous chems:
Sodium Hydroxide or NaOH
Mol. weight of ~**40 g/ mol**, need even more than in the first part.

Hydrochloric acid or HCl (aq). 36% HCl contains **11.64 mol/L**

DCM or dichloromethane for extraction, **K_2CO_3** (Potassium carbonate), **$MgSO_4$** (magnesium sulfate) to dry the DCM extracts. But one can make a brine out of plain NaCl instead of K_2CO_3 as well. **IPA** (isopropanol) to dissolve the freebase for the final salting.

EtOAc (ethyl acetate) and acetone to help precipitate and clean the final product.

Also consider **1,4-Dioxane** as a co-solvent for the reaction at a larger scale, which will help keep volumes more manageable.

"Quick" outline:

- From your amount in grams of MMDPPA prepare a 20 – 25x volume of 10% NaOH solution. So for **100 g amide you could use 2000 mL of 10% NaOH solution**. Or 200 g of NaOH dissolved in 2L of water. Let cool down or chill in the freezer.
    - An alternate method to look into for larger amounts would be to calculate from a **4 - 7x** hydride source or **NaOH** to amide counted by mol. So for **100 g amide use maybe 100 g NaOH**, but make a more concentrated solution. Say in 600 mL **water (~15%),** and then from this use a **1-2 x amount 1,4-Dioxane as water** or 600 mL to dissolve the amide, total volume would be now be **1200 mL**.
- Prepare a solution of NaDCC, use a bit more than 0.5 mol. eq. from the amide. So for **100 g amide use** ~0.242 mol or 62 – **62.4 g NaDCC dihydrate**. Use a max of 0.55x molar equivalent to prevent over-chlorination of the ring. Dissolve in about **3.9 x room temp. water in mL (250 mL)**. It helps to make sure that the NaDCC is a free flowing powder. So crush beforehand. If you cool the solution, crystals of NaDCC will start to grow in your beaker.
- Place a beaker with 2x or so the volume in an ice-salt bath (or $CaCl_2$). Begin **overhead stirring** of the amide, it will not dissolve in the water but will be in a **suspension**.
- Once the temperature is close to **5 °C**, add in the dissolved NaDCC in large portions. Add some water from cleaning out the beaker.
- Stir for about **6 – 8 hours in the cold**, bits of amide are floating around, but it is visibly less than in the beginning, in fact that is likely the insoluble cyanuric acid, the amide should have reacted and formed a N-chloroamide which would seem to dissolve better in water than the starting amide. Better to monitor by TLC this part of the reaction. Not so bad if temperature rises to 8 °C, 12 °C?.
- Continue cooling with more additions of ice.
- While still cool, filter off in a Büchner the formed cyanuric acid and transfer the solution to an addition funnel or large sep. funnel. Or gravity filter.
- Heat an excess of 10% or 15% NaOH to 80 °C beforehand, maybe in a round bottom flask so it heats quicker. For **100 g amide you could use 1.2 L 10% NaOH solution**.
- Slowly drip in the reaction mixture while it is at or below room temperature into the **80 °C – 90 °C** NaOH. Take your time dripping in slowly with good stirring for optimal yields. **Take a few hours**, stop dripping in if the temperature is too low: 75 °C, or too hot: 92 °C. In the beginning of addition, the reaction mixture is a **clear light orange-yellow**. But becomes cloudy and color changes with a bit more NaOH dripped in. Once all of the NaOH is added, continue heating at 75 °C for 90 minutes, then pour into a 4 L beaker to cool. The MDA freebase and impurities are **dark reddish brown blobs** that fall to the bottom of the beaker upon further cooling. Need to extract this.
- **Remember:** It seems that from what others have reported that both a longer time in the cold and a slower addition to hot NaOH, and additional time spinning in the heat help to increase yields.
- Let cool maybe to 30 °C then **extract with DCM** or chloroform in a 5L separatory funnel.. Maybe 3 or 4 extracts 2x 250 mL and 2x 200 mL for 100 g amide. Be careful shaking too hard with the first extract to prevent an emulsion. DCM layer will be dark yellow brownish and turbid.
- **Dry the DCM** with 2x 110 g **K_2CO_3 brine**. (Mix 220 g anhydrous K_2CO_3 in 200 mL water) Shake in a sep. funnel.
- **Dry further** in a large beaker with anhydrous $MgSO_4$. Maybe 10 – 15 g $MgSO_4$. Mix with magnetic stirring, and watch until it is free-flowing, but don't add much more than that to prevent product loss to the Epsom salts. DCM layer might still be cloudy at this point.

- Gravity filter directly into a pyrex plate set on top of a hot plate with a fan blowing. Once filtered the DCM layer should be a clear golden colour. Do so in a fume hood. Heat maybe to 60 °C, the DCM or chloroform should evaporate fairly quickly.
- Now there is the option to vacuum distill. Otherwise it might be possible to pick up the MDA with impurities now in IPA and form the hemisulfate salt, clean thoroughly, acid/base extract to a much cleaner freebase, pick up with IPA again and then form the final HCl salt. When forming MDA sulfate you avoid water by preparing 20% H_2SO_4 in IPA.
- Try to vacuum distill at **2.2 mbar with distillate coming over at about 137 °C**. Shulgin writes that the fraction collected boiled at 80 – 90 °C at 0.2 mm/Hg, this works out to 124.6 - 136 °C at 2.2 mbar.
- Use a cow adapter to only collect within this range, otherwise the distillate will not be pure.
- Distillate is clear but with a light tinge of yellow, yield on the distilled freebase should be in the 70 – 80% range. 85%? Weigh the MDA freebase oil and calculate mols.
- Calculate the amount of HCl, and dissolve in maybe 4 - 5x volume of IPA, then add very slightly diluted HCl (or even concentrated HCl if careful and slowly). **Optimal pH** would be **6.67** (pKA – 3). But suppose that anywhere between pH 6 and 7 is fine.
- Now there is an option to evaporate the IPA/ water/ MDA freebase mix on a hotplate with a fan, but depending on the amount it could some time. Shulgin dilutes in 2x the amount of diethyl ether to the IPA, to disrupt the polarity of the system. It is an anti-solvent. However EtOAc could work just as well, as could MTBE. After filtering and drying some, an option to mill in a coffee grinder or blender.
- Then I would chill and filter on the Buchner. Clean some more with EtOAc, and finally wash with a small amount of reagent grade ice cold acetone while sucking.
- Weigh the dry, white MDA and **rejoice**.

Procedure on 77.9 g MMDPPA:
Pretty much like in the outline above.

1) Calculate amide as **0.376 mol.**

2) Prepare 1950 mL of 10% NaOH, but use only 1530 mL to suspend the amide in a 4 L beaker. ~20x the amide. Cool to 6 °C with a $CaCl_2$ + ice + water bath. Use magnetic stirring.

3) Add dissolved NaDCC in a few large portions, pour in a bit more NaOH, and some more plain water from washing the NaDCC beaker. So actual total amount is closer to 1850 mL.

4) Stir for **8 hours, remove the cyanuric acid**, continue stirring under room temp for an additional 8 hours.

5) Heat **1L 10% NaOH** in a 6 L 3-neck flask on a heating mantle.

6) When the internal temperature is 80 °C, drip in from a 2 L sep. funnel the reaction mixture. Takes maybe 90 minutes or 2 hours to drip in between 75 and 92 °C, then spin for an additional 90 minutes. Transfer to a 4 L beaker and let cool to 30 °C.

7) Extract with 250, 250 mL DCM, and 200, 200 mL chloroform. Had run out of DCM so used chloroform.

8) **Dry the DCM** 2x with 110 g K_2CO_3 brine in a sep funnel, and then dry further with 10 g $MgSO_4$ in a beaker.

9) **Vacuum distill** between 1.9 and 4 mbar, collecting all fractions except for the very end. Distillate comes over between ~110 - 140 °C. Seems to be about 50 mL in the receiving flask, and I guess correctly that it should be about 50 g.

11) Weight of the distillate is 49.99 g or 0.279 mol MDA freebase or a **74% yield from the amide.** 53% from helional.

12) An **equimolar amount of concentrated HCl is 24 mL**. Dilute this amount to 50 mL, but probably no need for this. Mix the MDA freebase with about 250 mL IPA and neutralize that freebase with the HCl. Use a bit more than 24 mL.

13) Pour onto a plate, and set a fan to blow with heating. Crystals appear after an 30 minutes, but it takes a long time to fully evaporate. End up with orange - amber rocks. Colour is not to my liking.

14) Collect the still damp crystals, grind in a coffee grinder, then transfer to a Büchner funnel and wash with cool EtOAc.

15) Weigh and end up with **57.2 g** of the HCl or 0.265 mol. There is a bit more that slipped by when filtering yet to weigh.

16) With a Thiele tube, take a melting point test. Sample melts between **178 and 180 °C**. This is lower than reported values, but not by much, so there is a small amount of impurities. Color is not the whitest as well, but much better than initially collected. Another wash with cold acetone would result in a whiter MDA, but this one is perfectly fine for me.

17) Bio-assay 50 mg and it is surprisingly strong, much more fun than MDMA, really like 90s pills. Works as advertised.

Ponderings:
One would certainly attempt a more manageable liquid amount next and try the 1,4-Dioxane as the co-solvent on a 300 g helionalamide run. Another option to investigate is switching to a bromine source. Try and use NBS (N-bromosuccinimide) instead of NaDCC. It is reported to give high yields, but it would be mostly insoluble in cold water.

Part 2 Photos:

Set-up of for the reaction

Reaction in the cold continues, turns yellow

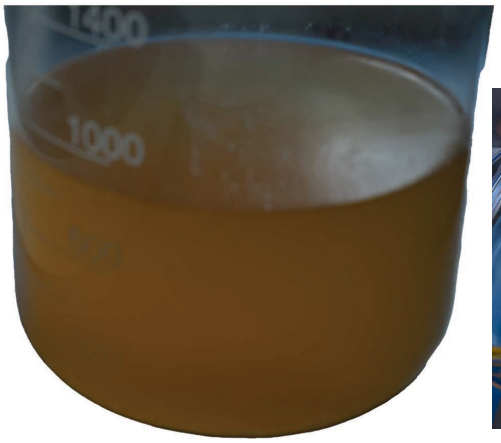
Decant mixture into another beaker, then gravity filter off the cyanuric acid. Note: colour is same in both beakers

Appearance of R.M. with N-chloroamide to be heated, transfer to 2 L sep funnel

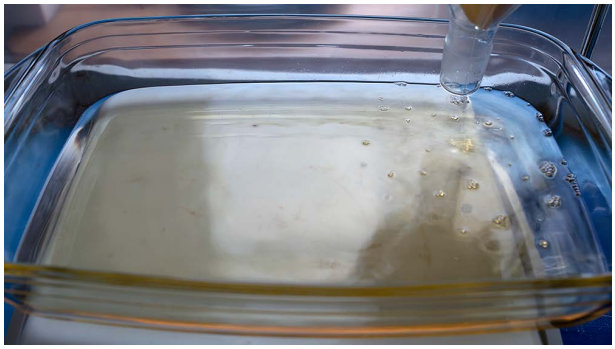
Reaction, dripping in from a seperatory funnel (not pictured)

When cooled, transfer to a broken 5 L sep. funnel and extract with DCM

Leftover basic wastewater to be discarded



After drying with both K_2CO_3 brine and $MgSO_4$, organic phase is still cloudy, not to worry!



Gravity filtering into a plate resolves turbidity. Heat and use fan to evaporate in a fumehood

Collect and prepare residue for distillation

Residual DCM boils off violently in the beginning then heat more, later cover with foil up to condenser

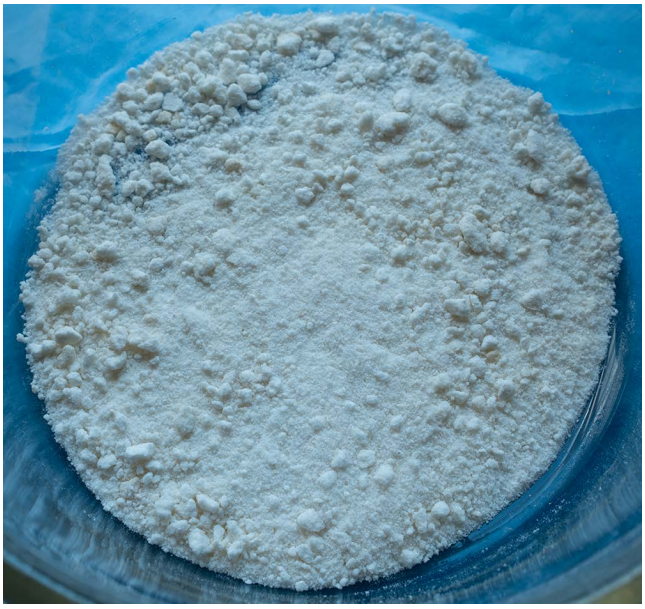
Distillate is clear, but does have a light yellow tint

Leftovers from distillation

Prepare for addition of HCl

Mist swirls when HCl is added to MDA

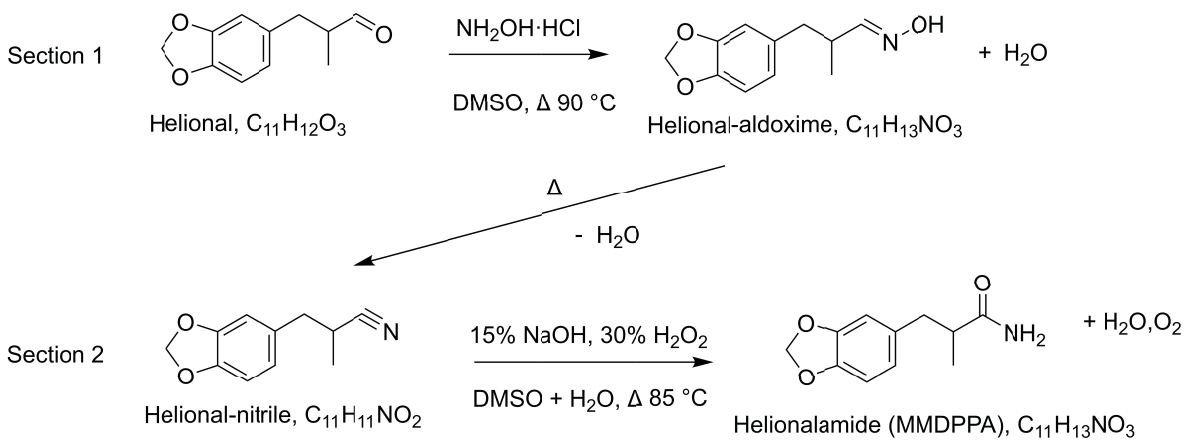
MDA HCl after IPA and water evaporates, not satisfied



Final Product cleaned with EtOAc. Looks better.

And finally, a general schematic of the reactions taking place. There is more than this going on, with transient molecules coming and going if one delves into the details. Looking into the actual electron transfers, and the real mechanism behind the one-pots, the Hofmann rearrangement in Section 2 of Part 2 as an example. There are a few more steps not shown below... Discover for yourself, get to the bottom of it.

Part 1



Part 2